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ANNOTATED BIBLIOGRAPHY OF BERYLLIUM

J. H. de Pruneda

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Deliverable for LL09-WPT080-PD04: Characterization of Beryllium Alloys

Jean H. de Pruneda

November 18, 2009

Publications reporting impurity concentrations

Al Goldberg: Be Manufacturing processes, UCRL-TR-222539, 2006

The author lists the major impurity for domestically-produced nuclear and structural grades of beryllium.

Rointan F. Bunshah: A Fresh Look at the Problems in Be Metallurgy, UCRL-6410 1961

The paper is mainly focused on the ductility of beryllium as a function of impurity content. The author includes a table with extensive impurities listed as a function of both supplier (Brush-Wellman and Pechiney) and production method (i.e., pebble, powder, induction melt, electron-beam melt, etc).

Yury M. Verzilov, Kentaro Ochiai, Satoshi Sato, Masayuki Wada, Michinori Yamauchi, Takeo Nishitani: Analysis of Impurities in Be affecting evaluation of tritium breeding ratio JAERI-Research 2004-2005

The publication lists impurities for several Brush-Wellman beryllium products that were manufactured in 1950 for assessment on how the impurity content affects the neutron absorption cross-section.

Dlrow Company <http://www.dlrow-cn.com/products/BERYLLIUM.htm>

Dlrow produces beryllium in two forms: metal ingots and powder. They give nominal impurity contents for both with the oxygen content being much higher in the powder than the ingot form. Iron and carbon impurities are slightly higher in the powder with the ingot having higher manganese, copper, magnesium and nickel contents.

D. S. Gelles: Post Irradiation Evaluation of Beryllium Pebbles, Final Report, Pacific Northwest National Laboratory

This paper gives an elemental analysis of impurities for 3 Brush-Wellman and one Japanese beryllium material. Grain sizes were generally larger in irradiated specimens with a low dislocation density. The microstructure changes are associated with the generation of helium bubbles. This gives rise

to highly non-equiaxed thin platelet-like cavities on the basal plain. Optical and SEM micrographs of pebbles are included.

Lungu et al: Beryllium Coatings on Metals,
www.ipp.mpg.de/eng/for/projekte/pfmc/pfmc.../I13_Lungu.pdf

The presentation is focused on coating various metal substrates with beryllium. However, there is a slide with elemental analysis of two different beryllium base materials: beryllium from pressed flakes from NFF Pitesti, and a bulk beryllium from UKAEA. The flake beryllium generally had greater impurities than the bulk beryllium, with the largest differences (i.e., greater amounts in the flake beryllium) with iron, chromium and manganese. The bulk beryllium had a greater silicon impurity content than the flake.

There are also some microstructures of deposited beryllium films.

Blosser et al: Analysis of Trace Impurities in High-Purity Be: UCRL-71886 1969

The report gives impurity analysis of a large number of samples (they claim most ever) using spark-source mass spec and gamma activation analysis. Impurity analyses are given for an unknown commercial grade, Pechiney S grade, Berylco, UKAEA, and Brush pebble and powder beryllium.

The authors divide the purification mechanisms and the associated impurities into three classes:

1. Those with vapor pressures higher than liquid or solid beryllium at elevated temperatures , e.g., sodium, potassium, chlorine, magnesium.
2. Those with vapor pressures lower than beryllium, e.g., iron, titanium, chromium.
3. Those which are partially or totally insoluble in liquid beryllium, e.g., BeO, Be₂C.

Gunther & Cook : Chemical Analysis of Be Shells, UCRL-TR-217909.

The authors analyzed NIF target shells using ICP-MS. Gives impurity analysis in as-provided ingot (PF-60) by Brush-Wellman.

Cliff W. Price: Ion microanalysis of Be, UCRL-98385 (1988)

The author found that **the results obtained by ion microanalysis correlate with the metallurgical processing history of beryllium** (*emphasis mine*). The relative concentrations of fluorine and chlorine can indicate whether fluoride or chloride salts were used in the initial reduction process used to produce the beryllium. Chlorine was significantly higher than fluorine in the

Electrofusion material, which indicates it was from material reduced from the chloride salt. The presence of carbon-containing particles in the Electrofusion material also could be related to the use of graphite molds in the reduction process. Both fluorine and chlorine as well as carbon were quite low in a specimen prepared by multiple vacuum arc melts and vacuum distillation, which attests to the effectiveness of that technique.

The copy of the report has poor-quality micrographs included.

Heiko Kleykamp: Quantitative X-ray microanalysis of beryllium using a multilayer diffracting J. Anal. At. Spectrom., 1999, 14, 377–380

The maximum solubility of impurities in annealed industrial Be heats is reported.

W. G. Boyle, C. H. Otto, Jr.: Precipitation of beryllium from homogeneous solution by decomposition of an acetylacetone species in basic solution, UCRL-70194, August 21, 1967

The authors include recipes for making Be, BeO and hydroxides and chemical analyses of products.

Bunshar: Impurity Removal by distillation of Be from the solid state, Lawrence Livermore National Laboratory, UCRL-12253, 1965

The report discusses the impurities in beryllium distilled from solid state. Impurities appear to be dependent on evaporation rate relative to beryllium.

“The metallic impurities present in Be may be classified according to their evaporation rates relative to Be as follows:

1. Evaporation rates much lower than Be, e.g., Fe, Ni, Si, Ti, Cr, Nb, etc.
2. Evaporation rates similar to Be, e.g., Al and Cu.
3. Evaporation rates much higher than Be, e.g., Mn, Ca, Mg, Na, etc.

Thus Fe, Ni, Si, Ti, and Cr would tend to concentrate in the distilland; Mn, Ca, Mg, Na, etc., would be almost completely removed from the distilland; and Al and Cu would behave in an intermediate fashion.”

R. F. Bunshah: The Effect of Purification on Some Mechanical Properties of Beryllium, Lawrence Livermore National Laboratory, UCRL-7522, April 3, 1964

The researcher investigated a large number of different beryllium products from a variety of sources, including A.E.R.E and U.S.S.R. The impurity content of all beryllium products is reported.

Microstructural Studies

Stuart I. Wright and James D. Cotton, Microtextural Characterization of a Be Weldments: "Textures and Microstructures", 1995, Vol. 23, pp. 7-19

Microstructural examination of a beryllium weldment. The authors conclude that grains grow in response to thermal gradient, not in response to the orientation relationship to other grains in weld. The authors use grain misorientation statistics to analyze the grain structures in the weld.

D. S. Gelles: Post Irradiation Evaluation of Beryllium Pebbles, Final Report, Pacific Northwest National Laboratory

This paper gives an elemental analysis of impurities for 3 Brush-Wellman and one Japanese beryllium material. Grain sizes were generally larger in irradiated specimens with a low dislocation density. The microstructure changes are associated with the generation of helium bubbles. This gives rise to highly non-equiaxed thin platelet-like cavities on the basal plane. Optical and SEM micrographs of pebbles are included.

Cliff W/ Price, and J. C. Norber (Evan & Associates): Analysis of Particles in Be by Ion Imaging, Lawrence Livermore National Laboratory, UCRL-100797

The authors analyzed powder-pressed Be (DOE Spec. 0001319) and SR-grade from Pechiney using SIMS. They then looked under TEM to correlate microstructure with impurity content/location. "The relative concentration of F and Cl can indicate whether fluoride or chloride reductions were used to produce the beryllium".

Cliff Price: Analysis of Low and High-Voltage SEM images of Beryllium, Proceedings of the XIIth International Congress for Electron Microscopy 1990

The researcher reports on ion beam damage in commercial grade beryllium sheet: prominent microtwins are visible. A fine faceted microstructure is formed by the ion beam. Machined commercial grade Be showed surface defects. Fracture surfaces were similar to machined surfaces.

Beryllium Use in Nuclear Reactors

T. A. Tomberlin: Beryllium – A Unique Material In Nuclear Applications, Nov 2004, INEEL/CON-04-01869 Preprint

The publication outlines the uses of beryllium in a nuclear reactor. The authors list some of the nuclear reactors around the world known to use

beryllium as a blanket, including the BR2 Belgian Engineering Test Reactor in Mol, Belgium, the MARIA research reactor of the Institute of Atomic Energy in Poland, the High Flux Isotope Reactor near Oak Ridge, Tennessee, and at Idaho National Engineering Environmental Laboratory, in their test reactor. The paper lists the 6 nuclear reaction chains of beryllium. All produce helium that causes swelling in the beryllium, producing internal stresses and changes to the mechanical properties of the beryllium.

Yury M. Verzilov, Kentaro Ochiai, Satoshi Sato, Masayuki Wada, Michinori Yamauchi, Takeo Nishitani: Analysis of Impurities in Be affecting evaluation of tritium breeding ratio JAERI-Research 2004-2005

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D. S. Gelles: Post Irradiation Evaluation of Beryllium Pebbles, Final Report, Pacific Northwest National Laboratory

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Dylst, Segher, Druyts, and Braet: Removing tritium and other impurities during industrial recycling of beryllium from a fusion reactor, 8th Intn'l conference on Tritium Science and Technology, Sept. 2007

Thirty-one reactors around the world use beryllium. The publications lists the places that manufacture beryllium for reactor applications – Russia (Kurchatov Inst., RIAR, Dimitrovgrad); USA (ORNL, BNL, INEL); Petten (?), Franca (Saclay), SCK.CEN?

The authors also describe the chlorination processes for purification and give a flow chart.

Oxidation of Beryllium

*C. Tomastik, W. Werner, H. Störi: Oxidation of Be by Auger microscopy
Institut für Allgemeine Physik, TU Wien, Wiedner Hauptstraße 8-10, 1040 Wien*

The authors investigated the diffusion of oxygen in beryllium to investigate its potential use as an oxygen getter in a nuclear reactor. The authors found that oxidation kinetics vary strongly between different batches of beryllium, suggesting impurities play an important role in oxidation kinetics. The authors also found that beryllium particles are loosened from the matrix when oxidized at 600C for 20 hours. The particles are micron-sized. The authors used sprayed and sintered materials. Unfortunately, the paper does not list an impurity analysis.

Sources of Beryllium

CIA: Production and Uses of Lithium and Beryllium in the Soviet Bloc, 1952. CIA Office of Reports, http://www.foia.cia.gov/browse_docs.asp?doc_no=0000316356

This declassified and sanitized report lists the production sites and major ore sites for beryllium in the former Soviet Union.

Hanafee, Musket & Stratman: High-purity Be foils, Lawrence Livermore National Laboratory, UCID-21847, Dec 1989.

The report describes the availability of beryllium sheet (for foils) in 1989. A recipe for making foils is included. The authors include a description of fluorescence analysis for oxygen.

McCarville et al: Technical Issues for Be Use in Fusion Blanket Applications: Lawrence Livermore National Laboratory, UCID-20319, Jan. 1985

The authors explore the implications of worldwide availability of beryllium and its use in nuclear power reactors, including the impact availability of beryllium will have on the economic decisions to reprocess and reuse irradiated beryllium. The report includes the 1985 estimates of beryllium by country.

Particle Dispersion

R. L. Baskett et al: Measurement and Dispersion Modeling of Beryllium from High Explosive Tests at Site 300 during 1991, UCRL-TM-229101 Lawrence Livermore National Laboratory, 2007

The authors report on particle dispersal from high-explosive dispersal experiments conducted at Site 300. The focus for the beryllium particles was the dispersal of respirable particles and whether or not they were capable of reaching the Site 300 boundary.

Corrosion of Beryllium

Jeffrey R. Friedman and James E. Hanafey: Corrosion/Electrochemistry of Monocrystalline and Polycrystalline Beryllium in Aqueous Chloride Environment UCRL-ID-137482

Corrosion studies as a function of pH of single crystal and polycrystalline beryllium. Different crystal orientations produce different corrosion rates. Included in the report are micrographs of corrosion pits. Older beryllium is more anisotropic than newer beryllium, so the age of beryllium needs to be considered when assessing corrosion of beryllium parts.

Mechanical Properties (including fracture)

Jankowski et al: Crystallization of Be-B metallic glasses, UCRL-JC-147-456

The paper outlines the research aimed at producing glassy-type beryllium targets for NIF. TEM and SADP micrographs of the evolution of nanocrystalline regions as a function of annealing temperature are included.

Grain size of beryllium produced by evaporation and sputter deposition can be refined with metal impurities. Adding B > 11% serves as glassy phase former. Then annealed and nanocrystalline structure evolves.

"It's generally accepted that the low ductility is associated with its hexagonal close packed (hcp) structure, localized slip, and high impurity content (especially that of BeO in Be powder products)."

Allen et al: Diamond-turning HP-21 Be to achieve an optical surface, Lawrence Livermore National Laboratory, UC-38, 1975

The authors concluded that beryllium cannot be diamond-turned to an acceptable optical finish. The tools wore out first. They used Kaweck HP-21 Be (Honeywell). Rod 1.5 in diameter, 3 in. long. YS=35ksi, elong. 2% in 1 in. grain size, <25 μm . The impurity contents are given. The researchers performed x-ray fluorescence to look at impurities. They concluded the inclusions were BeC, AlFeBe₄ and FeBe₁₁. They base this on the Mohr's hardness numbers for these compounds, and the x-ray fluorescence results. There are some poor-quality micrographs of beryllium finished surfaces.

Wadsworth and Nieh: Improved microyield strength in beryllium produced by Vapor Deposition, Lawrence Livermore National Laboratory, UCRL-JC-143493. Scripta met.

Good micrographs of PVD Be thick films. Also includes fracture surfaces.

Hanafee: EFFECT OF ANNEALING AND ETCHING ON MACHINING DAMAGE IN STRUCTURAL BERYLLIUM, Lawrence Livermore National Laboratory, UCRL-83303, submitted to J. Applied Metalworking

The report contains a discussion on machining damage and mechanical properties. Includes a discussion about prior processing and the effect on mechanical properties after machining. It might be possible to know prior processing even after annealing, etching, etc.

"In the present study, the optical appearance of the microstructure, the presence of a very high microhardness near the surface, and the response of the surface regions to annealing all indicate a highly worked surface layer. This evidence indicates that more serious consideration should be given to the possibility that a strain-hardened, oriented surface layer is a basic cause of low mechanical properties in machining- damaged beryllium."

Harris and Dunegan: Fracture Toughness of Beryllium, Lawrence Livermore National Laboratory, UCRL-70255, 1967. J. Mat. Research & Standards.

K_c values for Bruch N50A and S200 are given. The publication describes a technique to induce a precrack for fracture specimens. (Bad copy of report – several pages blank).

Paris and Harris: An Engineering Evaluation of the Status of Utilization of Beryllium from the Viewpoint of Fracture Mechanics, Lawrence Livermore National Laboratory, UCRL-72442, 1970

Review of classical fracture mechanics and available information in the literature for beryllium.

Tardiff, Kuhn, Kelly, Fracture Toughness of Sheet Be, Lawrence Livermore National Laboratory, UCRL-73714, 1972

The authors compare the fracture toughness of beryllium from ingot and powder beryllium. At the time the paper was written there were no standardized fracture toughness tests. The report includes some nice micrographs of microstructure in ingot sheet and powder sheet. There are also some great micrographs of fracture surfaces from fracture specimens.

Tardiff: Fracture Toughness of thin Be Sheet, Lawrence Livermore National Laboratory, UCRL-51544, 1974. (PhD thesis)

This is a PhD thesis and contains an extensive review of fracture mechanics, including a review of fracture properties and metallurgy of beryllium. Ingot sheet from Rocky Flats, and powder sheet from Kawecki Berylco Industries were investigated. Impurity analyses for these products are given.

Also included are micrographs of microstructure and fracture surfaces as a function of annealing temperature, TEMs of ingot sheet beryllium, as received and after annealing for one hour at various temperatures 680C – 830C. Also included are TEM micrographs of fracture specimens.

Steinberg: Experimental Program to Study the Mechanical Properties of Beryllium – A Status Report, Lawrence Livermore National Laboratory, UCID-18729 (1980)

This is an interim status report on gas-gun experiments to determine shock mechanical properties of beryllium.

Goldberg et al: Mechanical Properties Evaluation of some “Commercial Beryllium Materials: Lawrence Livermore National Laboratory. UCRL-87392, June 30, 1982

This paper was prepared for inclusion in the Proceedings of JOWOG-22, Golden, Colorado, April 19-20, 1982.

The report contains micrographs and SEM micrographs of fracture surfaces.

Velisavljevic et al: Structural and Electrical Properties of Be Metal to 66 GPA studied using Designer DACs, Lawrence Livermore National Laboratory, UCRL-JC-145825, Sept. 2001

The report documents results of Diamond Anvil Cell studies of beryllium. The authors found that the equation of state that they determined differs from that obtained from shock tests.

Steinberg: The Effect on work-hardening in dynamic deformation of beryllium, Lawrence Livermore National Laboratory, UCID-20243, 1984.

The report describes Hopkinson split bar experiments. No evidence of phase change up to 35 GPa was detected.

Steinberg, et al: Work-Hardening and Effective Viscosity in solid Beryllium, Lawrence Livermore National Laboratory, UCRL-91892, Physica, Volumes 139 & 140B, pages 762-765, 1986

The authors compare Hopkinson split bar, shock impact and cylinder deceleration experiments to hydrodynamic code simulations. By increasing the work-hardening in the Steinberg-Guinan constitutive model, excellent agreement between the experiments and calculations was reached.

Evans, et al: High-Pressure/High-Temperature Studies of Be, 2006, Lawrence Livermore National Laboratory, UCRL-TR-229496.

The authors present their preliminary results of high-temperature Diamond Anvil Cell studies of the phase diagram of beryllium at high temperatures and pressures.

Welding, Joining and Cutting of Beryllium

Hanafee and Ramos: Laser Fabrication of Be Components, Lawrence Livermore National Laboratory, UCRL-JC-121436, Sept, 1995

The report describes damage done to beryllium when machined conventionally – twins, microcracks, residual stresses and crystallographic texture lasting 100 microns in depth. Traditional welding on beryllium was performed with different filler material (Al-Si alloy). Old technique (>30 years in 1995). Authors developed laser cutting and welding techniques for beryllium sheet (0.5mm to 2.00 mm), beryllium alloy (62Be/38Al) and BeO composite. They used Nd:YAG and CO2 lasers. The authors also described autogenously laser-welded Be capsules.

John J. Truhan and Lawrence M. Wagner: Fabrication Thin Beryllium Windows for X-Ray Applications, Lawrence Livermore National Laboratory, UCRL-83767, Nuclear Instruments and Methods, Vol. 176 (3), Pp. 481-485, 1980

The authors describe a method of solid-state joining of beryllium to a substrate using PVD-deposited silver to minimize stresses in thin beryllium sheet for x-ray windows.

Landon, et al: Development of high-strength braze joints in Beryllium, Lawrence Livermore National Laboratory, UCRL-90294

The authors describe brazing of beryllium using aluminum with the goal of minimizing the aluminum thickness.

Knowles and Hazlett: High-Strength Low-Temperature Bonding of Beryllium and other Metals, Lawrence Livermore National Laboratory, UCRL-72342, 1970

The report has a discussion on the limitations of joining beryllium. The goal was to bond beryllium at temperatures below 600C. This could not be achieved without another intermediate metal (generally silver). It was also necessary to use a special cleaning technique on the surface prior to welding. The authors tried welding beryllium in a variety of atmospheres; air, O₂ and He.

Wright and Cotton: Microtextural Characterization of a Beryllium Weldment: Los Alamos National Laboratory, Los Alamos, NM 87545 (USA), Textures and Microstructures, 1995, Vol. 23, pp. 7-19

The researchers characterized a beryllium weld using grain-boundary misorientation. The report includes micrographs of the weldment.

Al Goldberg : Joining of Be, Lawrence Livermore National Laboratory, UCRL-TR-224718, September 26, 2006

The author presents an overview of joining techniques and weld designs for all beryllium types: alloys, thin and thick sheets, etc. Joining techniques include: welding, brazing, solid-state bonding, and soldering.

Eldon F. Westlund: Vacuum Furnace Brazing of Beryllium, Lawrence Livermore National Laboratory, UCRL-6391, March 1961

The report describes vacuum brazing techniques that have been developed for joining beryllium to titanium, stainless steel and to itself using silver as the filler. Shear strengths of about 20,000 psi were obtained for all three types of brazes. The report includes some nice photographs of brazed joints.

Richard J. Foley: Machining of Be with the LLNL Precision Engineering Research Lathe, Lawrence Livermore National Laboratory, UCID-20386, 1985.

The purpose of the study was to determine the optical properties of machined beryllium surfaces when prepared under highly controlled conditions using high-quality machine tools and CBN cutting tools. The work was performed in cooperation with Hughs aircraft. The report contains photograph of machined surfaces.

Processing

Bunshah and Juntz: PURIFICATION OF BERYLLIUM BY CRUCIBLE-FREE VACUUM MELTING AND DISTILLATION PROCESSES, Lawrence Livermore National Laboratory, UCRL-7591, August 1964

The authors classify impurities in beryllium with respect to:

1. Vapor pressure of impurity relative to beryllium
2. Solubility in liquid Be

Most impurities imparted by processing – i.e., from crucible, melt/crucible interactions and atmosphere. The authors describe a crucible-free process.

The report contains a discussion of various analytic techniques: mass spectrometry, gamma-ray activation analysis for O₂ and C, combustion carbon methods, and neutron activation analysis. The authors compared other analyses with mass spectrometry and concluded that mass spectrometry was a comparably good technique.

The beryllium used was SR grade Pechiney electrolytic flake and commercial-grade powder-metallurgy-type beryllium.

Dylst, Segher, Druyts, and Braet: Removing tritium and other impurities during industrial recycling of beryllium from a fusion reactor, 8th Intn'l conference on Tritium Science and Technology, Sept. 2007

Thirty-one reactors around the world use beryllium. The publication lists the places that manufacture beryllium for reactor applications – Russia (Kurchatov Inst., RIAR, Dimitrovgrad); USA (ORNL, BNL, INEL); Petten (?), Franca (Saclay), SCK.CEN?

The authors also describe the chlorination processes for purification and give a flow chart.

Kershaw: A Cause of Pitting in Beryllium, Lawrence Livermore National Laboratory, UCRL-87370

The report is a discussion of how pitting defects in chemical milling after machining are related to impurities introduced during processing.

Compilations

JOWOG 22B Be Meeting Presentations, CONF-9305224, 1993.

This is a compilation of JOWOG 1993 presentations. Included were:

- Status of commercial Be, presentations by Nuclear Materials Inc, Manufacturing Science Corp, and Brush-Wellman, Inc.
- Beryllium Technology in the Former Soviet Union
- Beryllium technology in India
- Presentations on new DOE guidelines for beryllium exposure.
- Microstructure and properties of rapidly solidified Be-Al alloys
- Al-Be alloy characterization
- Be-Al alloy development
- Hydrostatic extrusion of fine tubing
- Continuous fiber reinforcement using SiC and Al₂O₃ fibers
- Joining and mechanical properties of Be-Al alloys
- Recovery of beryllium Swarf
- Centrifugally atomized beryllium powders
- AWE work on atomized beryllium powders
- Plasma spraying of beryllium
- Direct Hot Isopressing of preforms
- Progress in determining the elevated temperature tensile properties of beryllium
- Properties of beryllium Bellville Washers
- High-temperature stress-strain behavior of beryllium

- Beryllium tensile testing using super grips
- Production of beryllium components using non-ozone depleting chemical
- Future of beryllium supply and beryllium technology
- DOE beryllium use
- Beryllium technology transfer

*D. Lane: Current Be Literature: A selected Bibliography January 1958-August 1959
Zanier 1959*

Contains extensive bibliography of work prior to 1959, by subject area.

Walsh, et al: Beryllium Chemistry and Processing, pub. ASM, 2009

A new encyclopedic publication covering almost every aspect of beryllium, from mining, to physical, chemical, mechanical properties, metallurgy to uses of beryllium.